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Nipecotic acid hydrochloride

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.022
wR factor = 0.057
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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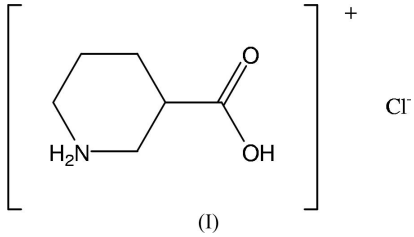
The title compound (3-carboxypiperidinium chloride), $C_6H_{12}NO_2^+\cdot Cl^-$, is the hydrochloride of nipecotic acid and is used as a drug intermediate and in the synthesis of γ-aminobutyric acid (GABA) uptake inhibitors. The geometric parameters are in the normal ranges. The crystal packing is stabilized by O—H···Cl and N—H···Cl hydrogen bonds.

Comment

Nipecotic acid or 3-piperidinecarboxylic acid is used as a drug intermediate and also in the synthesis of γ-aminobutyric acid (GABA) uptake inhibitors (Muralidhar *et al.*, 1994). A review on the neurochemical and behavioural profile of a derivative of nipecotic acid hydrochloride has been reported by Suzdak & Jansen (1995). In view of the importance of nipecotic acid, the present paper reports the crystal structure of nipecotic acid hydrochloride, (I).



(I)

A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; Mogul Version 1.0.1; Allen, 2002). The heterocycle adopts a chair conformation. The crystal packing is stabilized by O—H···Cl and N—H···Cl hydrogen bonds.

Experimental

Nipecotic acid was purchased from the Aldrich Chemical Company and was converted to its hydrochloride by adding a mixture of isopropyl alcohol and hydrochloric acid (80/20). The compound was recrystallized from ethanol.

Crystal data

$C_6H_{12}NO_2^+\cdot Cl^-$
M_r = 165.62
Monoclinic, $P2_1$
a = 7.2545 (10) Å
b = 7.2018 (9) Å
c = 7.7886 (13) Å
β = 97.819 (12)°
V = 403.14 (10) Å^3
Z = 2
D_x = 1.364 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8363 reflections
θ = 3.8–25.7°
μ = 0.42 mm^{-1}
T = 173 (2) K
Block, colourless
0.37 × 0.23 × 0.19 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.861, T_{max} = 0.925
3354 measured reflections
1477 independent reflections
1460 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.5°
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.057
S = 1.10
1477 reflections
104 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.0946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.075 (10)
Absolute structure: Flack (1983), 671 Friedel pairs
Flack parameter: 0.10 (6)

Table 1
Selected geometric parameters (Å, °).

C2—N3	1.4972 (18)	C7—O2	1.187 (2)
N3—C4	1.502 (2)	C7—O1	1.323 (2)
C2—N3—C4	113.78 (12)	O2—C7—C1	123.39 (16)
O2—C7—O1	122.79 (15)	O1—C7—C1	113.68 (14)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···Cl1	0.89 (3)	2.11 (3)	2.9985 (13)	173 (2)
N3—H3A···Cl1^{i}	0.96 (2)	2.20 (2)	3.1428 (15)	166 (2)
N3—H3B···Cl1^{ii}	0.90 (2)	2.47 (2)	3.2525 (15)	147 (2)
N3—H3B···O2^{ii}	0.90 (2)	2.42 (2)	2.9590 (19)	119 (2)

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+1$; (ii) $x, y, z-1$.

All H atoms were located in a difference map. Those bonded to C atoms were positioned geometrically and refined with fixed individual displacement parameters (set to 1.2 times U_{eq} of the parent C atom) using a riding model, with C—H = 0.99 and 1.0 Å for secondary and tertiary H atoms. The H atoms bonded to N and O atoms were refined freely. The *ADDSYM* routine in *PLATON* (Spek, 2003) detects a pseudo-centre of symmetry in the structure, which is fulfilled by approximately 80% of the structure. This would mean changing the space group from $P2_1$ to $P2_1/m$. In this case, the molecule must lie on a mirror plane. However, the molecule does not have any symmetry at all. Therefore, $P2_1$ is the correct space group and it is just a pseudo-centre of symmetry that *PLATON* detects.

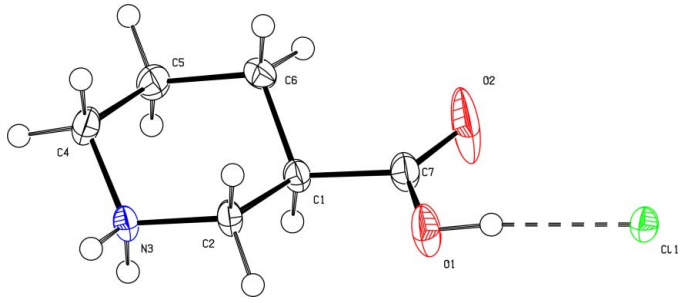


Figure 1
Perspective view of the title compound, showing the atom numbering and displacement ellipsoids drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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